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INTERNATIONAL APPLICATION PUBLISHED UNDER THE PATENT COOPERATION TREATY (PCT)

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(21) International Application Number: PCT/SE98/01940 (22) International Filing Date: 27 October 1998 (27.10.98) (30) Priority Data: 9704013-3 3 November 1997 (03.11.97) SE (71) Applicant (for all designated States except US): ASEA BROWN BOVERI AB [SE/SE]; S-721 83 Västerås (SE). (72) Inventors; and (75) Inventors/Applicants (for US only): JOHANSSON, Tommy [SE/SE]; Lupinvägen 2, S-722 46 Västerås (SE). KORSKE, Håkan [SE/SE]; Bomansgatan 7, S-722 20 Västerås (SE). (74) Agents: BERGLUND, Stefan et al.; Bjerkéns Patentbyrå KB, Östermalmmsgatan 58, S-114 50 Stockholm (SE).		(81) Designated States: AL, AM, AT, AT (Utility model), AU, AZ, BA, BB, BG, BR, BY, CA, CH, CN, CU, CZ, CZ (Utility model), DE, DE (Utility model), DK, DK (Utility model), EE, EE (Utility model), ES, FI, FI (Utility model), GB, GE, GH, GM, HR, HU, ID, IL, IS, JP, KE, KG, KP, KR, KZ, LC, LK, LR, LS, LT, LU, LV, MD, MG, MK, MN, MW, MX, NO, NZ, PL, PT, RO, RU, SD, SE, SG, SI, SK, SK (Utility model), SL, TJ, TM, TR, TT, UA, UG, US, UZ, VN, YU, ZW, ARIPO patent (GH, GM, KE, LS, MW, SD, SZ, UG, ZW), Eurasian patent (AM, AZ, BY, KG, KZ, MD, RU, TJ, TM), European patent (AT, BE, CH, CY, DE, DK, ES, FI, FR, GB, GR, IE, IT, LU, MC, NL, PT, SE), OAPI patent (BF, BJ, CF, CG, CI, CM, GA, GN, GW, ML, MR, NE, SN, TD, TG). Published <i>With international search report.</i> <i>In English translation (filed in Swedish).</i>
(54) Title: A METHOD FOR PRODUCING A MATERIAL, A COMPOSITION AND A USE OF THE COMPOSITION (57) Abstract <p>A method of producing a material, according to which method a particulate polymer is mixed with a particulate, inorganic hydroxide, and the mixture is treated in such a manner that it forms a solid body. Before said treatment the polymer is given an average particle size in the order of 500 μm or less. The invention also concerns a composition comprising the particulate polymer and the particulate, inorganic hydroxide, where the average particle size of the polymer is in the order of 500 μm or less. The composition is used for producing a solid body suitable to constitute a component in a power electronics device.</p>		

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- 5 Method for producing a material, a composition and use of
the composition

BACKGROUND OF THE INVENTION AND PRIOR ART

10 The present invention concerns a method for producing a
material, according to which method a particulate polymer is
mixed with a particulate, inorganic hydroxide and the
mixture is treated in such a manner that it forms a solid
body.

15 The invention also concerns a composition, comprising a
particulate polymer and a particulate, inorganic hydroxide,
which composition is suitable to be treated in such a manner
that it forms a solid body. The invention also concerns a
20 use of said composition.

As a way of description but not limiting purpose the
invention will be described with reference to HVDC-
applications (HVDC = High Voltage Direct Current), that is
25 applications where a strong direct current voltage is
present and where it is desired that components, arranged
close to current conducting conductors and other electrical
equipment, are both insulated and fire resistant. A typical
example of such an application is the stations or plants
30 where alternating current which is conducted in leads on
land is converted to direct current in order to, for
example, with a sea-cable be conducted in water, for example
a longer distance across a large straight or the like.

35 In such stations or plants there are a large number of
components with different tasks but with the common feature

that they preferably are of an insulating kind. Furthermore,
it is extremely important that they have very good fire
resistant properties, since a fire in such a plant would
have devastating consequences caused by thereby arisen power
5 failures, for example cutting off the light in whole cities.

An example of such a component is the cooling water tubes
which exist in such a plant. For the above mentioned reasons,
it is extremely important that these, when they are exposed
10 to strong heat or a flame of fire, does not catch fire.

Such tubes are often made of polymer materials, since these
materials by reasons of manufacture are suitable for
producing tubes and moreover may be brought to have
15 preferable physical and mechanical characteristics. A
problem is however the fire resistant properties of such
materials. It is desirable that these materials fulfil the
classification UL V0, which is the best fire class according
to Underwriter's Laboratories. The classification is done in
20 that a vertically positioned test rod of the material is lit
in its lower end with a bunsen flame under given conditions.
The burning time after the bunsen flame has been removed is
measured. The classification UL V0 means in principle that
the test rod is extinguished within 5 seconds and that no
25 burning drops fall from the rod. There are extremely few
thermoplastic polymer materials which have such fire
resistant properties in combination with the other physical
and mechanical characteristics which such a tube should
have. A known material which as such is fire resistant and
30 which has satisfactory mechanical characteristics is
polyvinylidene fluoride, PVDF, which above all is
characterised by a high resistance to chemicals and flame
proofness at an elevated temperature. However, should such a
fluoride plastic material like PVDF be exposed to such a
35 strong outer influence by heat or fire from an external fire
source that it is set fire to, it has the disadvantage that

it then emits poisonous, strongly corrosive gases. Further disadvantages are its high density and high price. For want of better alternatives PVDF has, however, been used in tube applications of the mentioned kind.

5

With other polymers, such as polyethene, PE, which does not have as good fire resistant properties as PVDF, it is necessary to add a flame inhibiting means to the polymer itself in order to achieve fire resistant properties. The
10 flame inhibiting means may be an inorganic hydroxide with the capacity to split to form water when it is exposed to high temperatures. A known example of such a hydroxide is aluminiumtrihydrate, ATH. Its good flame inhibiting effects have been known for long. However, for conventional
15 production of fire resistant components including for example polyethene and ATH as main components, a very high content of ATH (about 40-60% by weight) is required in order for the material to achieve the best fire classification. This results in particular in a strongly reduced ductility
20 of the material, which is not desirable for tube applications. In the conventional production a granulate of the polymer is mixed with a powder of the inorganic hydroxide and is thereafter treated by for example hot pressing or extrusion with the purpose of forming the
25 desired component, for example a tube.

It if were not for the reduced ductility of the material, a mixture of polyethene and aluminiumtrihydrate would be suitable for the mentioned tube applications, since
30 polyethene is an inexpensive material and since polyethene as well as ATH are advantageous materials from an environmental point of view. In order to achieve a sufficiently high ductility, with the present method of production, such low contents of ATH in the material are
35 required that the fire resistant properties cannot fulfil the requirements called for by authorities and others in

connection with for example HVDC-applications. This can be said to be the case generally for materials which principally comprise a polymer which is made fire resistant by means of addition of an inorganic hydroxide.

5

SUMMARY OF THE INVENTION

A purpose of the present invention is thus to present a method of producing a material which comprises a polymer and
10 an inorganic hydroxide, wherein the material has good fire resistant properties thanks to the inorganic hydroxide, while the hydroxide is present in such a low content in the material that it does not dramatically influence the mechanical characteristics, in particular the ductility.

15

This purpose is achieved by the method as initially defined, characterised in that the polymer, before said treatment, is given an average particle size in the order of 500 micrometers or less. Thereby an essentially improved mixture
20 of the inorganic hydroxide in the polymer is obtained compared to when methods of production of the conventional kind are used, by which a polymer granulate with an average particle size of 1-3 mm is used. Due to the improved mixture of the inorganic hydroxide a relatively seen higher flame
25 inhibiting effect is achieved at a given content of the inorganic hydroxide in the material. A smaller amount of the inorganic hydroxide than for a conventional production is thus required in order to achieve corresponding fire resistant properties of the material. The smaller amount of
30 used inorganic hydroxide leads in its turn to an improved ductility of the material, which is desirable in particular for the production of such components as cooling water tubes in HVDC-plants.

35 Concerning the mixture which is used in the method according to the invention the following should be noted: preferably

the mixture does not comprise any larger amounts of any other materials than the particulate polymer and the inorganic hydroxide. However, smaller amounts of other materials may be included, for example small amounts of known stabilizers for the polymer may be included in the mixture (preferably in the form of antioxidants). Such stabilizers or antioxidants may already be included in the particulate polymer or may be added to the mixture before or during the formation of the solid body. Suitably such further material is included only to a very small part, for example some percentage by weight.

According to another preferred embodiment, the polymer is given an average particle size in the order of 250 μm or less, preferably 100 μm or less and most preferred 25 μm or less. As the average particle size is made smaller a correspondingly improved distribution of the inorganic hydroxide is achieved in the particulate polymer, and a correspondingly smaller amount of inorganic hydroxide is required for achieving the required fire resistant properties. The advantageous distribution of the inorganic hydroxide in the polymer presupposes of course that the hydroxide has a relatively small average particle size. The use of fine particulate inorganic hydroxides is however not new by itself, but is usual in conventional methods of production, where the inorganic hydroxide which is used most often is present in powder form with a very small average particle size. In this case its average particle size is equal to or less than that of the polymer, preferably in the order of 10 μm or less.

According to a further preferred embodiment, the polymer is a thermoplastic resin. Such plastics are generally more suitable than thermosetting resins for the suggested method, since thermosetting resins suitably are arranged in the form of a liquid resin and a liquid thermosetting agent and are

not in the same manner as thermoplastic resins suitable for treatments such as hot pressing or extrusion of for example tubes.

- 5 According to another preferred embodiment, the polymer is a polyolefin. Polyolefins are particularly suitable for the method according to the invention and are also often both inexpensive and environmentally friendly.
- 10 According to another preferred embodiment, the polymer is polyethene. Polyethene is inexpensive, harmless to the environment, and has physical as well as mechanical characteristics which make it very well suited to be extruded to and used as for example tubular products.
- 15 According to another preferred embodiment of the method, the inorganic hydroxide is of the kind which, when it is heated to a certain temperature, is split to form water. Thereby the risk is further reduced that the material, when it is
- 20 exposed to strong heat or to a flame, will catch fire.
- According to another preferred embodiment, the inorganic hydroxide comprises aluminiumtrihydrate as main component. Aluminiumtrihydrate is very suitable as a fire-inhibiting
- 25 component in a polymer based material. It may be produced in the form of a powder with a small average particle size and has, furthermore, the advantage that it is harmless to the environment and not poisonous for humans.
- 30 According to another preferred embodiment, the treatment comprises subjecting the mixture to a mechanical force at an elevated temperature. A typical example of such a treatment is hot pressing. Another example is extrusion.
- 35 According to another preferred embodiment, the solid body, which is formed by the treatment, is subjected to electron

radiation. By electron radiation it is possible to crosslink for example polyethene, which gives this material further improved mechanical characteristics. Normally, when the content of an inorganic hydroxide, such as ATH, is high, there are considerable difficulties to obtain any crosslinking worth mentioning in a polymer material such as polyethene by electron radiation. The method according to the invention allows, however, a low content of the inorganic hydroxide, whereby crosslinking by electron radiation is made possible.

According to a further preferred embodiment, the proportion of inorganic hydroxide in the mixture is less than 40% by weight, preferably less than 25% by weight, suitably between 5 and 20% by weight.

A further purpose with the invention is to provide a composition comprising a particulate polymer and a particulate, inorganic hydroxide, which composition is suitable to form a solid body which is to a large extent fire resistant due to the inorganic hydroxide, but where the latter is present at such a low content that it does not to any noteworthy extent influence the physical and mechanical characteristics of the body. For this purpose a good distribution of the inorganic hydroxide in the composition is required. The composition should be such that it makes such good distribution possible.

This purpose is achieved by a composition as initially defined, which is characterised in that the particulate polymer has an average particle size in the order of 500 μm or less.

As described above in connection with the method, the composition may also contain very small parts of other

materials, for example stabilizers for the polymer in the form of antioxidants.

According to a preferred embodiment, the average particle
5 size is in the order of 250 μm or less, preferably 100 μm or less, and most preferred 25 μm or less. It has been found that a smaller average particle size of the polymer results in a better distribution of the inorganic hydroxide and in that the latter therefore may exist at a lower content at
10 the same time as acceptable fire resistant properties are maintained for the body which is formed from the composition.

According to a preferred embodiment, the polymer is a
15 thermoplastic resin, preferably a polyolefin and most preferred polyethene. The reasons for this choice of material have already been mentioned in connection with the description of the method according to the invention.

According to a preferred embodiment, the inorganic hydroxide
20 is of the kind which, when it is heated to a certain temperature, is split to form water. Preferably, the inorganic hydroxide comprises aluminiumtrihydrate, ATH, as main component. The advantages with this choice of material
25 have already been described in the description of preferred embodiments of the method according to the invention.

According to a preferred embodiment, the proportion of
inorganic hydroxide in the composition is less than 40% by
30 weight, preferably less than 25% by weight, suitably between 5 and 20% by weight.

Another purpose of the invention is to define a use for
which the composition according to the invention is
35 particularly advantageous. One such use is the production of

a solid body, suitable to constitute a component in a power electronics device.

5 Preferably this use concerns a use where the component in question is a tube suitable for allowing a cooling medium to run therethrough.

10 Another suitable use of the component is for electric apparatuses, in particular for electrical apparatuses which are used within a low voltage range (up to about 1000 volt). In such electrical apparatuses, the component in question may be used both as a material of construction and as an insulator.

15 EXAMPLE

A very fine particulate polyethene powder with a particularly advantageous average particle size (average particle size \approx 20-40 μ m) is mixed with a fine particulate
20 aluminiumtrihydrate, ATH in powder form with an average particle size in the order of 1-3 μ m. The composition formed thereby was pressed at a moderate temperature (in the order of 150°C). A solid body was thereby obtained. This body was exposed to electron radiation, such that crosslinked
25 polyethene was formed.

The amount ATH in the composition was only in the range of 12% by weight. In spite of this comparatively low content of ATH, the formed body obtained fire resistant properties
30 according to the class UL V0 for a 9 mm thick test rod, that is the best fire classification according to Underwriter's Laboratories. The result may be explained in that the fine particulate form of the polymer, in this case the polyethene, gives a very good mixture of and thereby a good
35 distribution of the aluminiumtrihydrate, ATH, in the composition. Thereby the efficiency of the ATH becomes much

higher. The low content of ATH, on the other hand, brought about that the ductility of the body was only influenced to an insignificant degree and was close to that of pure polyethene. It could also be established that a relatively
5 high degree of crosslinking was obtained in the polymer, in this case about 73%, which may also be attributed to the small influence which ATH has on the composition when it is present at such a low content as in this case.

10 Of course, a number of varieties and alternative embodiments of the invention will be obvious to the person skilled in the field without this person thereby departing from the invention. The scope of protection of the invention is defined primarily by the appended main claims.

15 Furthermore, it is presupposed that the given average particle sizes are valid for particle or powder batches where the particle size distribution is of a normal kind for this technical field and where any important deviations from
20 the average particle size in individual particles therefore do not exist at all or exist only exceptionally.

As a further example of an inorganic hydroxide with the ability to split to form water, magnesium hydroxide may
25 additionally be mentioned.

Claims

1. A method of producing a material, according to which
5 method
- a particulate polymer is mixed with a particulate inorganic hydroxide, and
 - the mixture is treated in such a manner that it forms a solid body, characterised in that the polymer,
10 before said treatment, is given an average particle size in the order of 500 micrometers or less.
2. The method according to claim 1, characterised in that the polymer is given an average particle size in the order
15 of 250 micrometers or less.
3. The method according to claim 1, characterised in that the polymer is given an average particle size in the order of 100 micrometers or less.
20
4. The method according to claim 1, characterised in that the polymer is given an average particle size in the order of 25 micrometers or less.
- 25 5. The method according to anyone of the claims 1-4, characterised in that the polymer is a thermoplastic resin.
6. The method according to anyone of the claims 1-4, characterised in that the polymer is a polyolefin.
30
7. The method according to anyone of the claims 1-4, characterised in that the polymer is polyethene.
8. The method according to anyone of the claims 1-7,
35 characterised in that the inorganic hydroxide is of the kind

which, when it is heated to a certain temperature, is split to form water.

9. The method according to anyone of the claims 1-8,
5 characterised in that the inorganic hydroxide comprises aluminiumtrihydrate as main component.

10. The method according to anyone of the claims 1-9,
10 characterised in that the treatment comprises subjecting the mixture to a mechanical force at an elevated temperature.

11. The method according to anyone of the claims 1-10,
15 characterised in that the treatment comprises one of pressing or extrusion.

12. The method according to anyone of the claims 1-11,
characterised in that the solid body is subjected to electron radiation.

20 13. The method according to anyone of the claims 1-12,
characterised in that the proportion of inorganic hydroxide in the mixture is less than 40 percentage by weight.

25 14. The method according to claim 13, characterised in that the proportion of inorganic hydroxide in the mixture is less than 25 percentage by weight.

15. The method according to claim 13, characterised in that the proportion of inorganic hydroxide in the mixture is
30 between 5 percentage by weight and 20 percentage by weight.

16. Composition comprising a particulate polymer and a particulate, inorganic hydroxide, which composition is suitable to be treated in such a manner that it forms a
35 solid body, characterised in that the particulate polymer

has an average particle size in the order of 500 micrometers or less.

17. Composition according to claim 16, characterised in
5 that the particulate polymer has an average particle size in the order of 250 micrometers or less.

18. Composition according to claim 16, characterised in
10 that the particulate polymer has an average particle size in the order of 100 micrometers or less.

19. Composition according to claim 16, characterised in
that the particulate polymer has an average particle size in the order of 25 micrometers or less.

15 20. Composition according to anyone of the claims 16-19, characterised in that the polymer is a thermoplastic resin.

21. Composition according to anyone of the claims 16-19,
20 characterised in that the polymer is a polyolefin.

22. Composition according to anyone of the claims 16-19, characterised in that the polymer is polyethene.

25 23. Composition according to anyone of the claims 16-22, characterised in that the inorganic hydroxide is of the kind which, when it is heated to a certain temperature, is split to form water.

30 24. Composition according to anyone of the claims 16-23, characterised in that the inorganic hydroxide comprises aluminiumtrihydrate as main component.

25. Composition according to anyone of the claims 16-24,
35 characterised in that the proportion of inorganic hydroxide

in the composition is less than 40 percentage by weight, preferably less than 25 percentage by weight.

26. Composition according to claim 25; characterised in
5 that the proportion of inorganic hydroxide in the composition is between 5 percentage by weight and 20 percentage by weight.

27. Use of a composition according to anyone of the claims
10 16-24 for the production of a solid body, suitable to constitute a component in a power electronics device.

28. Use according to claim 27, characterised in that the
15 component is a tube suitable for allowing a cooling medium to run therethrough.

INTERNATIONAL SEARCH REPORT

International application No.

PCT/SE 98/01940

A. CLASSIFICATION OF SUBJECT MATTER

IPC6: C08K 3/32, C08J 3/20, C08L 23/06

According to International Patent Classification (IPC) or to both national classification and IPC

B. FIELDS SEARCHED

Minimum documentation searched (classification system followed by classification symbols)

IPC6: C08K, C08J, C08L

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched

SE,DK,FI,NO classes as above

Electronic data base consulted during the international search (name of data base and, where practicable, search terms used)

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C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
X	EP 0751184 A2 (ATOHAAS HOLDING C.V.), 2 January 1997 (02.01.97), page 3, line 38 - line 54, claim 1, abstract --	1-5,8-9
X	File WPI, Derwent accession no. 94-129458, Hitachi Cable Ltd: "Flame resistant insulated electric wire - coated with compsn. contg. specified polyolefin and high mol. wt. polyethylene, with magnesium hydroxide", JP,A,6076644, 940318, DW9416 --	16-18,20-24

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Date of the actual completion of the international search

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INTERNATIONAL SEARCH REPORT

International application No.

PCT/SE 98/01940

C (Continuation). DOCUMENTS CONSIDERED TO BE RELEVANT

Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
A	File WPI, Derwent accession no. 82-20990, Mitsubishi Petrochemical Co Ltd: "Inorganic filler-contg. polyolefin compsn. - comprising thermoplastic polyolefin matrix and dispersed phase of impact-absorbing polymer mixed with inorganic filler", JP,A,57023642, 820206, DW8211 --	16-26
A	EP 0004017 A1 (AEG-TELEFUNKEN KABELWERKE AKTIENGESELLSCHAFT), 19 Sept 1979 (19.09.79) -- -----	1-28

INTERNATIONAL SEARCH REPORT
Information on patent family members

International application No.
PCT/SE 98/01940

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